EFFECT OF UNTREATED AND ALKLAI TREATED ANACARDIUM OCCIDENTALE NUT SHELL BIOCHAR ON PHYSIO-CHEMICAL, THERMAL, MECHANICAL, ELECTRICAL AND SURFACE EROSION PROPERTIES OF VINYL ESTER COMPOSITES

A SYNOPSIS

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1. INTRODUCTION

Biochar-based composites are materials that combine biochar with other substances to enhance their properties for various applications. Biochar itself is a stable, carbon-rich product derived from the pyrolysis of organic materials, such as agricultural waste, under limited oxygen conditions. When integrated into composites, biochar can improve the mechanical strength, thermal stability, and environmental sustainability of the resulting materials.

In the context of a circular economy, biochar-based composites hold significant importance due to their ability to contribute to waste valorization, carbon sequestration, and resource efficiency. By converting organic waste into biochar, these composites help close the loop on waste streams, reducing the need for virgin materials and minimizing landfill usage. Additionally, the carbon stored in biochar can remain sequestered for centuries, mitigating greenhouse gas emissions and contributing to climate change mitigation. The use of biochar-based composites spans various industries, including agriculture, construction, and environmental remediation. In agriculture, these composites can enhance soil fertility and water retention, promoting sustainable farming practices. In construction, they can be used to create building materials with lower environmental footprints and improved durability. In environmental applications, biochar composites can aid in the removal of contaminants from soil and water, supporting ecosystem restoration and pollution control.

In recent days, the biochar from agricultural waste has been mixed with polymer matrices to develop composite structures for automobile components, aircraft components, construction materials, and decorative works. The surface treatment of biochar enhance the compatibility between polymer matrix and the biochar filler. Alkali treatment is an appropriate solution to remove surface contaminants and smoothen the highly porous carbon particles present in biochar. In the current research work, cashew (Anacardium occidentale) nut shells (AONS) were utilized to produce biochar through a slow pyrolysis process at 600°C. Various proportions (5, 10, and 15 wt.%) of untreated and 5% alkali-treated AONS biochar were incorporated into the vinyl ester matrix, and composite specimens were prepared. The Vinyl ester Resin /AONS and VER/alkali-treated AONS (a-t AONS) biochar composites were characterized using FTIR, XRD, TGA, tensile testing, flexural testing, impact testing, dynamic mechanical

analysis, and electrical conductivity measurements to analyze their potential for various applications.

2. LITERATURE SURVEY

Literature survey provides the fundamental concepts, methodology, testing procedures, properties and end use of fabricated composite specimens by previous researchers. The technical information gathered from previous literatures will enlighten the researcher's idea in to real product. In the current research the literatures related to natural fiber reinforced composites, effect of biochar on polymer composites, effect of chemical treatment on biochar on polymer composites are reviewed meticulously.

Sundarakannan et al. [1] prepared the polyester resin-based composites reinforced with red mud filled pineapple fiber at 10, 20, 30 & 40 % Wt. This investigation concluded that 10Wt % red mud composition, low erosion is achieved by an impingement angle of 60^o, an erodent velocity of 72 m/s, and an erodent outflow of 2.5 g/m^2. According to Rani et al. [2] the Hybrid powder fillers (WC + Ta/NbC) reinforced epoxy resin based composites showed the high thermal stability and low erosion rate. The erosion rate was measured at different impingement angle such as 30^o, 60^o, and 90^o. The hybrid GE composite possessed a higher thermal stability up to 522 ^oC. At $\alpha = 30^o$, the hybrid composite exhibited the lowest rate of erosion (Ws = 1.55 X 10^{-4} g/g). The date palm waste biochar was prepared at different pyrolysis temperature (300, 400, 500, 600, and 700 ^oC) and mixed with polypropylene matrix and composite specimens were prepared by Elnour et al. [3]. Das et.al [4] investigated the mechanical properties of four different type of biochar reinforced polypropylene based composites. The Rice husk, coffee husk, coarse wool, and landfill wood based biochar were prepared by pyrolysis process of temperature around 900 ^oC. Among four types of biochar reinforced composites, land fill wood biochar loaded PP composites exhibited the maximum tensile strength of 35.8 MPa, tensile modulus of 4.7 GPa, flexural strength of 67.2 MPa and flexural modulus of 4.4 GPa. The various concentration (5, 10 and 15wt.%) of cashew nutshell biochar added with unsaturated polyester resin and its mechanical and erosion properties were analysed by Sundarakannan et al. [5]. The author claimed that 10% cashew nut shell biochar filled polyester composite exhibited the maximum tensile, flexural, impact and hardness strength. The mechanical properties of composites are slightly decreased with 15wt. %

of filler loading. Richard et al. [6] investigated the erosion properties of various concentrations (0.5, 1, 1.5,2 and 2.5wt. %) of Rice husk biochar infused polyester resin. According to experimental results the specific wear rate and coefficient of friction reduced to 56.36% and 46.42% for composite specimen with 2.5 wt% filler loading and particle size of 45 nm. The electrical properties of Miscanthus plant biochar was analysed for different pyrolysis temp of 650, 700 and 750 °C. Based on research conducted by Giorcelli et al. [7] the carbon content increased with the pyrolysis temperature. Due to high carbon content, the composite with high biochar loading (15wt. %) possess better electrical conductivity (12.45 S/m) compared to pure epoxy plates (0.031 S/m).

Ray et al. [8] analysed the mechanical properties of different proportions of Raw and NaOH treated jute fibres (8, 15, 23, 30, and 35%) filled Vinyl ester based composites. Authors claimed that the flexural strength of 35% of alkali-treated jute fiber mixed vinyl ester composites increased by 16.65% compared to untreated fiber-loaded composites. Thiruchitrambalam et al. [9] investigated the mechanical properties of Raw and NaOH treated Roselle fiber (0, 10, 15, 20, 25, 30, 25, 40, 45 and 50wt.%) reinforced vinyl ester composites. The test results showed that a 40% fiber composition is optimal and exhibits superior tensile properties. The tensile strength and tensile modulus of VE/40% alkali-treated jute fiber increased by 62.86% and 25.92%, respectively, compared to VE/40% untreated jute fiber composites. Corn stover biochar and satin weaved sisal fibre (1 to 5%) / vinyl ester composites was investigated by Sivamurugan et al. [10]. The results showed that the hybrid vinyl ester composites exhibited maximum Tensile. Flexural and impact strength values at 5% of biochar loading. The corn stover biochar / sisal fibre / vinyl ester composites also possess hydrophobic properties. Ramraji et al. [11] analysed the mechanical properties of Almond shell biochar mixed with untreated and alkaline treated flax fibre (Biochar wt. % 5, 10 and 15wt. %) / vinyl ester composites. The test findings showed that the 10% biochar loaded composite with flax fibre that had been alkaline-treated had greater impact, flexural, and tensile strengths of 7.99 J, 79.5 MPa, and 71.5 MPa, respectively.

From the detailed literature review it has been evidence that the biochar filler acted as a strengthening agent for polymeric composites. The mechanical properties, thermal and erosion properties of polymer composites were enhanced with the influence carbonaceous biochar.

3. RESEARCH GAP

Based on literature survey the following research gap was identified

- Numerous studies have revealed that the use of particle filler and natural fibre as reinforcement filler in polymer matrix composite. Not much study has been done on using biochar as a reinforcing filler for polymer composites, even though fly ash, metal oxides, natural fibers, and inorganic fillers have all been extensively investigated.
- To increase the desirable characteristics of natural fibers, many researchers have performed surface enhancement techniques. The Biochar reinforced polymer composites also prepared by various researchers. Very few alkali-treated biochar particles have been used in polymer composites; in particular, treated biochar with vinyl ester composites has not been reported so far.
- Researchers have revealed the mechanical properties of a unique biochar derived from the cashew (Anacardium occidentale) nut shell reinforced polymer composite. No indications of the creation of untreated and alkali-treated cashew (Anacardium occidentale) nut shell (AONS) biochar studded vinyl ester composites have been documented.
- Furthermore, no articles on the solid particle erosion characteristics of treated and untreated ANOS biochar-filled vinyl ester composites have been published.

The data mentioned above, which came from a survey of the literature, serve as the stepping for this research.

4. RESEARCH OBJECTIVES

- To remove the cashew nut shells - a plant waste - from the ripe cashew apples. The pyrolysis method was used to create biochar from cashew (Anacardium occidentale) nut shells.
- To enhance the functionality, porosity structure, and surface topography of cashew (Anacardium occidentale) nut shell biochar, treated with 5% NaOH.

- To create vinyl ester-based polymer composites using the solution casting process in varying weight percentage (5%, 10%, and 15 wt %) of untreated and alkali-treated AONS biochar.
- To investigate the physical-chemical, morphological, thermal, mechanical, and electrical characteristics of vinyl ester composites using untreated and alkali treated cashew (Anacardium occidentale) nut shell biochar.
- To assess the visco-elastic characteristics of untreated and alkali treated ANOS biochar-loaded composites.
- Using synthetic untreated and alkali-treated cashew nut shell char biochar reinforced vinyl ester composite samples, to conduct erosion investigations.
- To examine the salient characteristics of multiple compositions (5, 10 and 15 weight percent) of vinyl ester composites infused with untreated and alkali treated AONS biochar.

5. MATERIALS AND METHODS

The commercially available unsaturated vinyl ester resin and the hardener materials such as methyl ethyl ketone peroxide (MEKP), cobalt naphthenate (CoNaph), and dimethyl aniline utilized in this work were supplied by GVR Enterprises, Madurai, Tamil Nadu, India. The cashew nut shells are collected from the local market, and it is washed and dried in an oven at 50 °C for 12 hrs. The dried cashew nut shell particles were placed in the air-tight (zero oxygen) container inside the pyrolysis reactor at the temperature of 600 °C. Further, pyrolyzed cashew nut shell fragments were processed in a mechanical planetary ball mill.

The methodology used in the present work is shown in Figure 1 To prepare untreated and alkali-treated Anacardium occidentale (cashew nut) shell biochar reinforced vinyl ester composites, the following methodology was employed: First, cashew nut shells were collected, cleaned, and dried before being pyrolyzed at 500 °C in an inert atmosphere to produce biochar. For alkali treatment, the biochar was soaked in a 5% sodium hydroxide (NaOH) solution for 4 hours, thoroughly washed with distilled water to remove any residual NaOH, and then dried at 50 °C for 12 hours. Both untreated and treated biochars were ground and sieved to obtain a uniform particle size. Vinyl ester resin and a suitable amount of methyl ethyl ketone peroxide (MEKP) as a

catalyst were mixed, followed by the gradual addition of the biochar filler (untreated or alkali-treated) with continuous stirring to ensure homogeneous dispersion. The mixture was then degassed to remove air bubbles and poured into pre-prepared molds. The composites were cured at room temperature for 24 hours, followed by post-curing at 80 °C for 3 hours. The cured samples were demolded and cut into specified dimensions for various characterization tests to evaluate their physico-chemical, thermal, mechanical, electrical, and viscoelastic properties. The composite fabrication is shown in Figure 2.

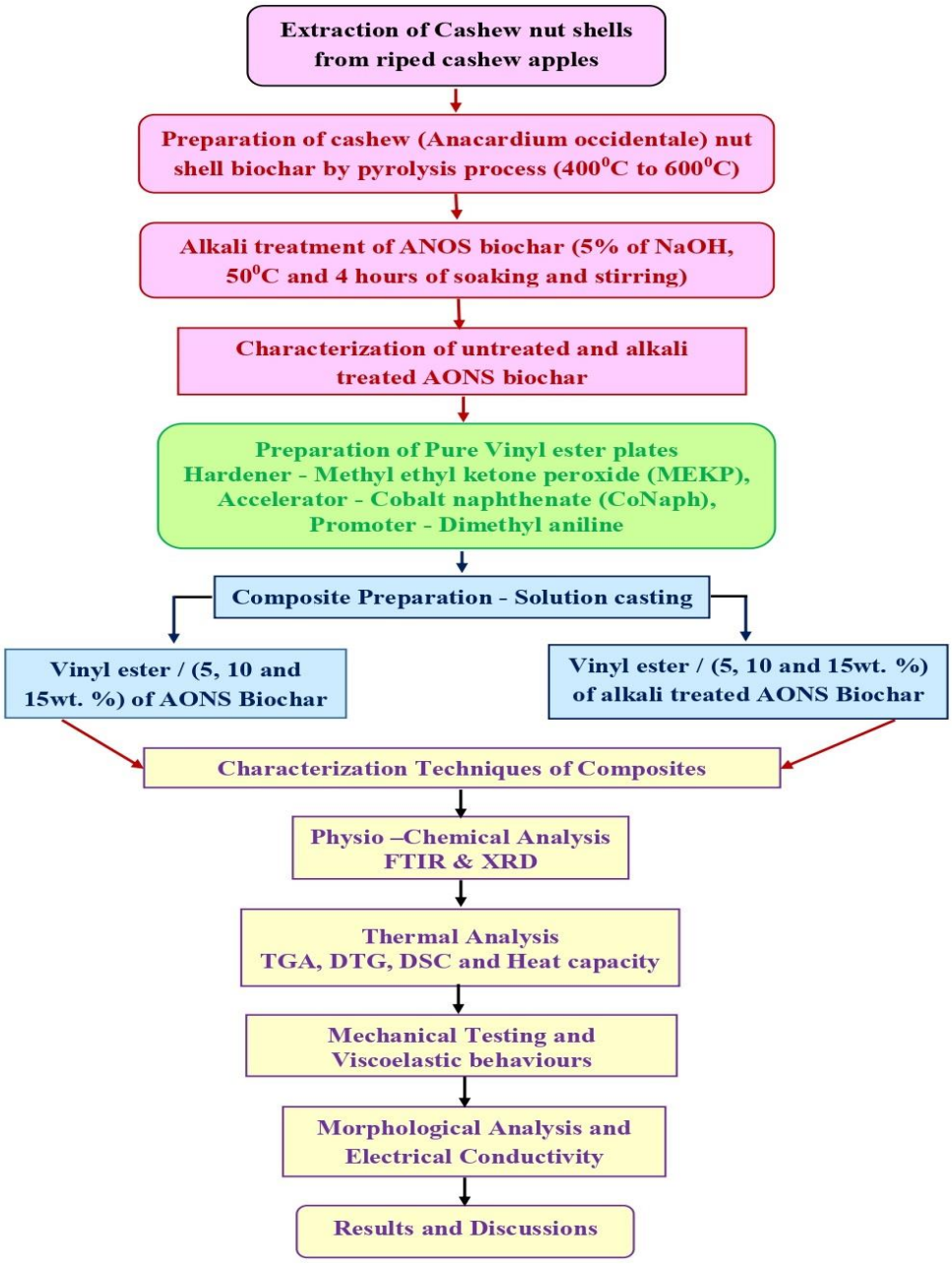
Extraction of Cashew nut shells from riped cashew apples
Preparation of cashew (Anacardium occidentale) nut shell biochar by pyrolysis process (400^{0}C to 600^{0}C)
Alkali treatment of ANOS biochar (5% of NaOH, 50^{0}C and 4 hours of soaking and stirring)
Characterization of untreated and alkali treated AONS biochar
Preparation of Pure Vinyl ester plates
Hardener - Methyl ethyl ketone peroxide (MEKP),
Accelerator - Cobalt naphthenate (CoNaph),
Promoter - Dimethyl aniline
Composite Preparation - Solution casting
Vinyl ester / (5, 10 and 15wt. %) of AONS Biochar
Vinyl ester / (5, 10 and 15wt. %) of alkali treated AONS Biochar
Characterization Techniques of Composites
Physio –Chemical Analysis
FTIR & XRD
Thermal Analysis
TGA, DTG, DSC and Heat capacity
Mechanical Testing and Viscoelastic behaviours
Morphological Analysis and Electrical Conductivity
Results and Discussions

Figure 1 Proposed Methodology

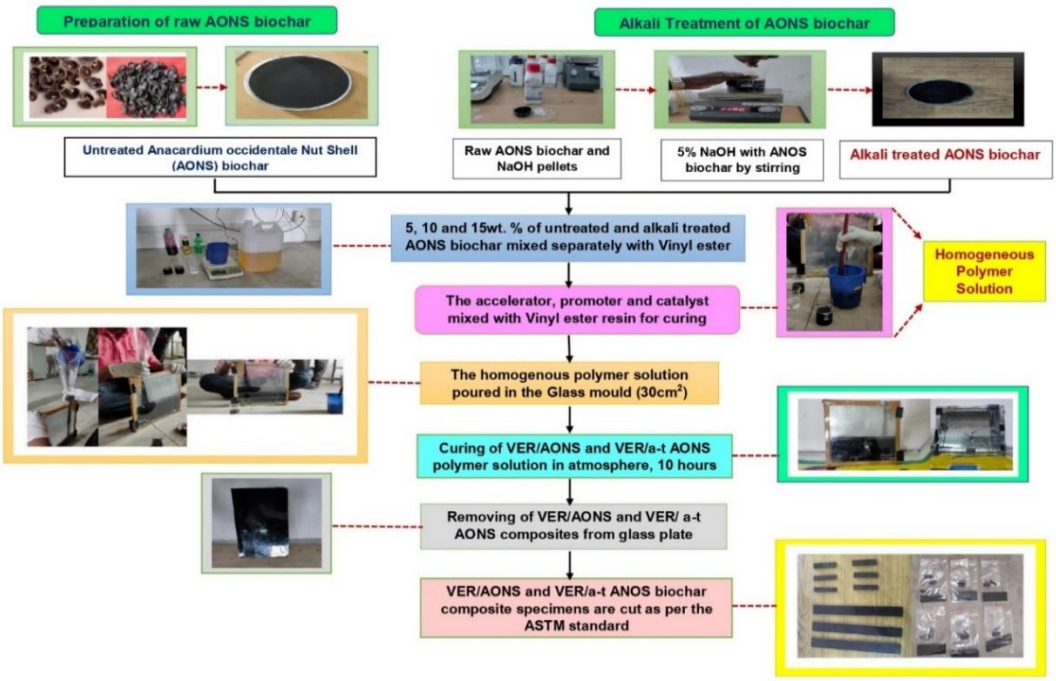
Preparation of raw AONS biochar
Alkali Treatment of AONS biochar
Untreated Anacardium occidentale Nut Shell (AONS) biochar
Raw AONS biochar and NaOH pellets
5% NaOH with ANOS biochar by stirring
Alkali treated AONS biochar
5, 10 and 15wt. % of untreated and alkali treated AONS biochar mixed separately with Vinyl ester
Homogeneous Polymer Solution
The accelerator, promoter and catalyst mixed with Vinyl ester resin for curing
The homogenous polymer solution poured in the Glass mould (30cm²)
Curing of VER/AONS and VER/a-t AONS polymer solution in atmosphere, 10 hours
Removing of VER/AONS and VER/ a-t AONS composites from glass plate
VER/AONS and VER/a-t ANOS biochar composite specimens are cut as per the ASTM standard

Figure 2 Preparation of AONS biochar, Treatment of biochar and Composite Fabrication

6. CHARACTERIZATION TECHNIQUES

The specifications of equipment's used to validate the properties of various proportions (5%, 10% and 15wt.%) of untreated and alkali treated AONS biochar reinforced vinyl ester composite is listed in Table 1.

Table 1 Experimentation of VE/AONS and VE/a-t AONS biochar composites

S.No.	Characterization Technique	Specifications	Properties to be measured
1	Particle size tester	Malvern Particle Size Analyzer (Range : 0.3 µm – 400 µm)	To find the particle size of the untreated and alkali treated Anacardium occidentale nut shell powder (AONS)
2	Surface Morphology	Field Emission Scanning Electron Microscope (Model: ZEISS SUPRATM 40)	To investigate the elemental composition of untreated and alkali-treated AONS biochar, VE/AONS and VE/ a-t AONS biochar composites
3	Fourier Transform Interferometry (FTIR)	FTIR analyzer Nicolet 5700, scanning range of 4000 to 400 cm^{-1}	To analyze the Chemical functional groups present in untreated and treated AONS biochar , VE/AONS and VE/a-t AONS biochar composites

4	X-ray diffraction analysis	Bruker AXS D8 Advance, Germany) with Cu K radiation and 2θ ranging from 5° to 60° at 5°/min	To determine the Crystallinity index of untreated and treated AONS biochar, VE/AONS and VE/ a-t AONS biochar composites.
5	Thermal Analysis	Thermogravimetric analyzer (Model: DTG-60H Shimadzu), Kyoto, Japan	To validate the thermal degradation behaviour of test samples.
6	Dynamic Mechanical Analysis	Dynamic mechanical analyzer (DMA Q800)	To evaluate the viscoelastic properties of fabricated composites.
7	Tensile and Flexural test	Universal testing machine (Tinius Olsen Hounsfeld H100 KS Series, Salfords, UK) Tensile test - ASTM standard D3039 - 200×20×3mm Flexural test - ASTM standard D790 - 125×13×3mm	To determine the stress and modulus of all the fabricated composites plates.
8	Impact Test	IZOD impact testing machine ASTM standard D256 of dimension 65×13×3mm.	To determine the hardness of pure vinyl ester plate and composite plates.
9	Electrical Properties	Plexiglass cylinder method Samples size 6x30x3 mm^3	To assess the electrical conductivity of all treated, untreated AONS biochar and composite specimen.
10	Solid particle erosion test	Air jet erosion tester TR-47 Ducom make model ASTM G-76 , specimen size 25×25×3mm	To determine the erosion rate of pure vinyl ester specimen and composite specimen against erodent discharge at different impingement angle

7. RESULTS AND DISCUSSONS

7.1 Physio-chemical properties of VER/AONS and VER/a-t AONS biochar composites

The FTIR analysis of VE/ANOS biochar and VE/ a-t AONS biochar composites revealed that there is no formation of new functional chemical functional groups as well as compatibility between the matrix and biochar filler improved by alkali treatment. Due to incorporation alkali treated AONS in vinyl ester matrix the shift of -OH bonds occurred and also enhance molecular integration between matrix and reinforcement observed. The similar observations was noticed by Das et al. [12]. The changes in wave number and chemical functional groups of different composition (5%, 10% and 15wt. %) of VE/ANOS and VE/ a-t AONS biochar listed in Table 2.

The XRD analysis of composites samples ensured that the incorporation of carbonized biochar content in the vinyl ester matrix the crystallinity index was improved. The physical structure of vinyl ester based composites plates are enhanced due to better compatibility of base materials. The crystallinity index of VE/15% ANOS biochar and VE/15% a-t AONS biochar composites are 32.8% and 48.4% respectively.

Table 2 FTIR Peaks of untreated and alkali treated AONS biochar filled VER composites

Wave numbers cm^{-1}							
VER/untreated AONS composites			**Assignment**	**VER/alkali treated AONS composites**			**Assignment**
5%	**10%**	**15%**		**5%**	**10%**	**15%**	
2932.89	2927.89	2929.62	O-H stretching	2941.44	2937.89	2932.89	O-H stretching
2861.60	2853.05	2853.05	N-H stretching -amine	2861.60	2825.96	2853.05	N-H stretching – amine
1904.27	1886.45	1860.08	C-H bending	1850.81	1833.70	1833.70	C-H bending (aromatic compound)
1691.85	1691.85	1700.40	C=O stretching	1753.86	1753.86	1753.86	C=O Stretching (carboxylic acid)
1470.16	1460.89	1505.09	C-H bending (alkane)	1682.58	1700.40	1691.85	C=N stretching (imine/oxime)
1257.62	1239.20	1239.20	C-O stretching	1470.16	1478.74	1478.79	C-H bending (methylene groups)
1035.33	1026.78	1008.96	C-N Stretching (amine)	1266.29	1257.02	1257.02	C-O stretching (alkyl aryl ether)
769.45	743.07	751.62	C-H bending	1044.60	1026.78	1035.33	S=O Stretching (sulfoxide)
				760.89	751.62	760.89	C=C bending (alkene)

7.2 Mechanical Properties of VER/AONS and VER/a-t AONS biochar composites

The mechanical properties of fabricated composite plates were analysed by calculating its tensile strength, flexural strength, impact strength as well as tensile and flexural modulus. The mechanical properties of VER/AONS biochar and VER/a-t AONS biochar composites are highly influenced with the addition of carbon-based biochar. The 15 wt. % of filler content showed maximum mechanical properties among other combinations (5 and 10%). The alkali treated AONS biochar loaded composites exhibited better tensile, flexural and impact properties compared to untreated AONS biochar loaded composites. The Tensile, Flexural and impact properties of VER/AONS

biochar and VER/a-t AONS biochar loaded composites shown in Figure 3, Figure 4 and Figure 5 respectively.

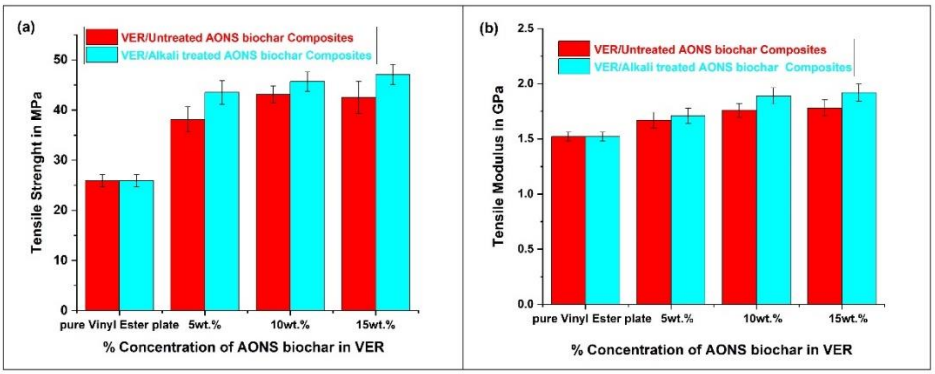

Figure 3 a) Tensile stress of VER/AONS and VER/a-t AONS biochar composites, b) Tensile modulus of VER/AONS and VER/a-t AONS biochar composites

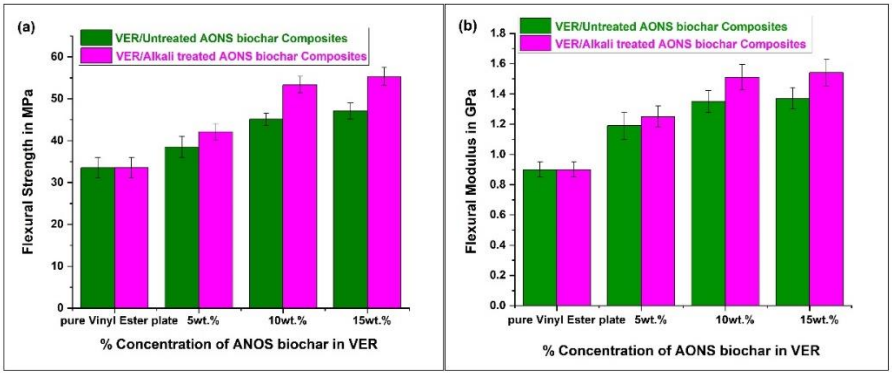

Figure 4 a) Flexural stress of VER/AONS and VER/a-t AONS biochar composites, b) Flexural modulus of VER/AONS and VER/a-t AONS biochar composites

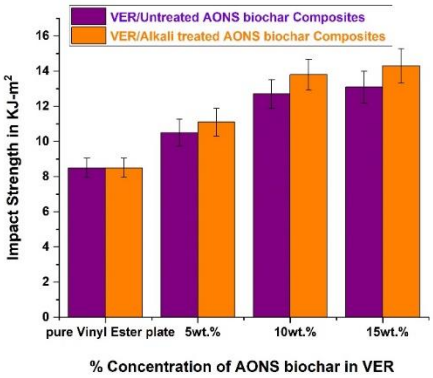

Figure 5 Impact strength of VER/AONS and VER/a-t AONS biochar composites

7.3 Thermal Analysis of VER/AONS and VER/a-t AONS biochar composites

According to graphical representations (Figure 6) of Thermal analysis, the thermal degradation temperature of composites increased with the incorporation of carbon based biochar filler Latha et al. [13]. The maximum degradation temperature of pure vinyl ester plate is 310 ^{o}C. The alkali-treated AONS biochar is tightly bonded with the intermolecular chains of vinyl ester. Thus, the thermal capacity of the alkali-treated AONS biochar composite is superior to that of the untreated biochar-loaded composite. The maximum degradation temperature of the VER/15 wt. % AONS biochar composite is 427.50 °C, while the VER/15 wt. % a-t AONS biochar composite has a maximum degradation temperature of 433.40 °C. Furthermore, the enhanced thermal stability of the composites with high filler content has been verified indirectly by the char yield, which grew from 6.7% (VE plate) to 44.11% (VER/15 wt.% AONS biochar) and 58.56% (VER/15 wt.% a-t AONS biochar). Additionally, the DSC plots demonstrated that the influence of the biochar content raised the melting and crystallisation temperatures of the composite plates.

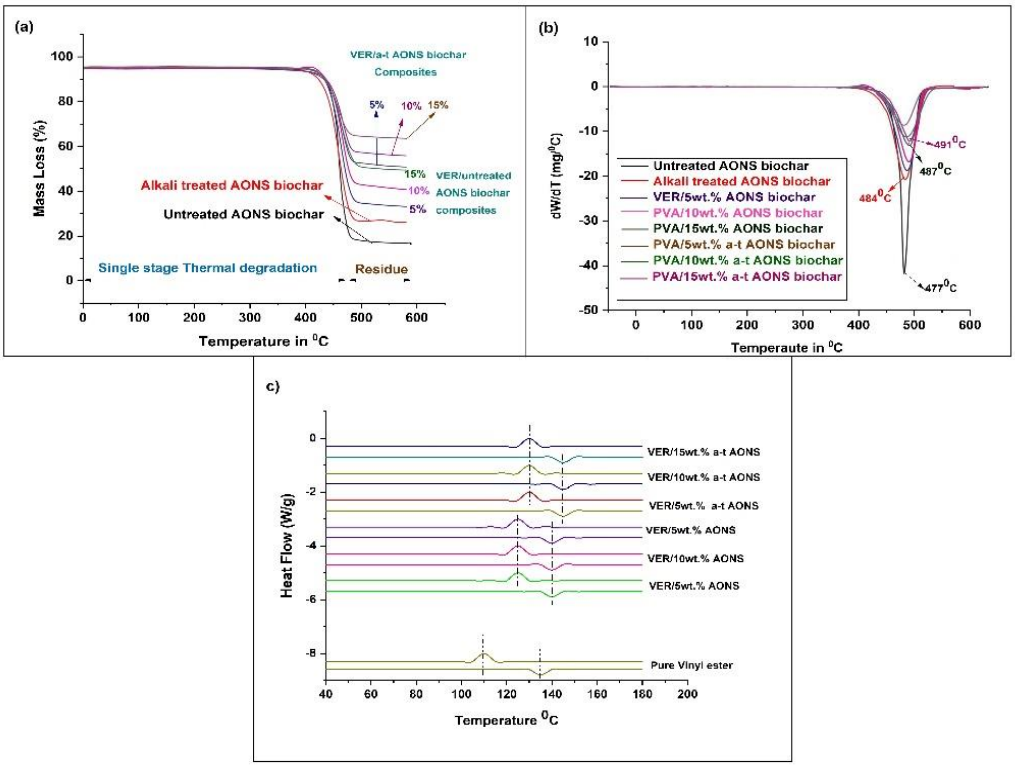

Figure 6 Thermal properties of VER/AONS and VER/a-t AONS biochar composites (a) TGA, (b) DTG and (c) DSC

7.4 Dynamic Mechanical Analysis of VE/AONS and VE/a-t AONS biochar composites

The storage modulus relates to the materials ability to store energy elastically. Similarly, the loss modulus (G" or E") of a material is the ratio of the viscous (out of phase) component to the stress, and is related to the material's ability to dissipate stress through heat, Akaluzia et al [14]. The untreated and alkali treated ANOS biochar loaded vinyl ester composites exhibited the increase in glass transition temperature corresponding to the increase in wt. % of filler content from 5% to 15wt.%. The thermal stress transfer at glass transition temperature also improved with the addition of biochar. The same findings also observed by Pudełko et al. [15]. The alkali treated AONS biochar loaded samples exhibited higher storage modulus and loss modulus compared to untreated composite samples. Figure 7 shows the variation of storage modulus and loss modulus corresponding to different composition of untreated and alkali treated AONS biochar in vinyl ester matrix.

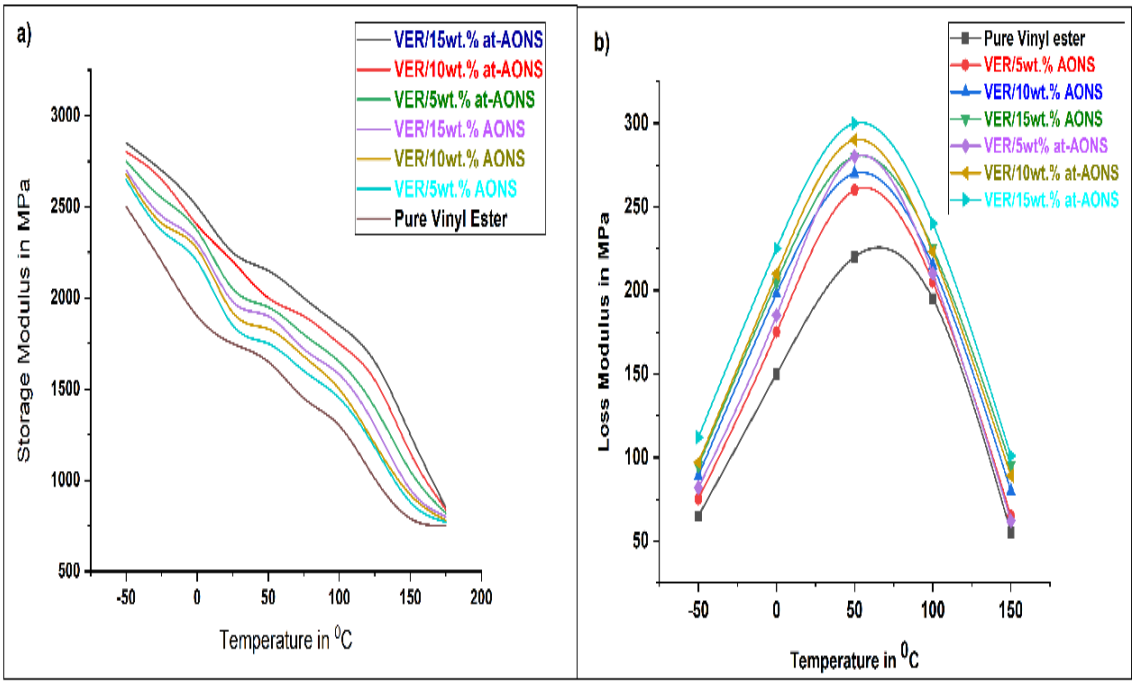

Figure 7 Viscoelastic properties (a) Storage modulus of VER/at AONS biochar composites (b) Loss modulus of VER/a-t AONS biochar

7.5 Electrical properties of VER/AONS and VER/a-t AONS biochar composites

The electrical conductivity is an important properties of composites which has been used in semiconductor devices. Based on the previous literatures the biochar possess an electrical conductivity range of 0.002 to 18.51 S/cm approximately.

The pyrolysis temperature is an important factor to improve the electrical conductivity of biochar. The carbon fiber filled composite materials showed moderate electrical conductivity reported by Minugu et al [16]. Typically, carbon fibres have an electrical conductivity of about 10^2 S/m, which is relatively low. Nonetheless, the electrical conductivity of carbon fibres can be improved by adding certain compounds. In the current research the untreated and alkali treated AONS biochar mixed with vinyl ester at different proportions (5, 10 and 15wt. %). The maximum pyrolysis temperature for preparation of AONS biochar is 600^0C. The electrical conductivity of untreated and alkali treated AONS biochar is 98.59 S/m and 128.94 S/m respectively. The pure vinyl ester specimen doses not conduct the electricity. The electrical conductivity of VER/15wt. % AONS biochar and VER/15wt. % a-t AONS biochar composites are 0.025S/m and 0.041 S/m respectively.

7.6 Solid particle erosion test of VER/AONS and VER/a-t AONS biochar composites

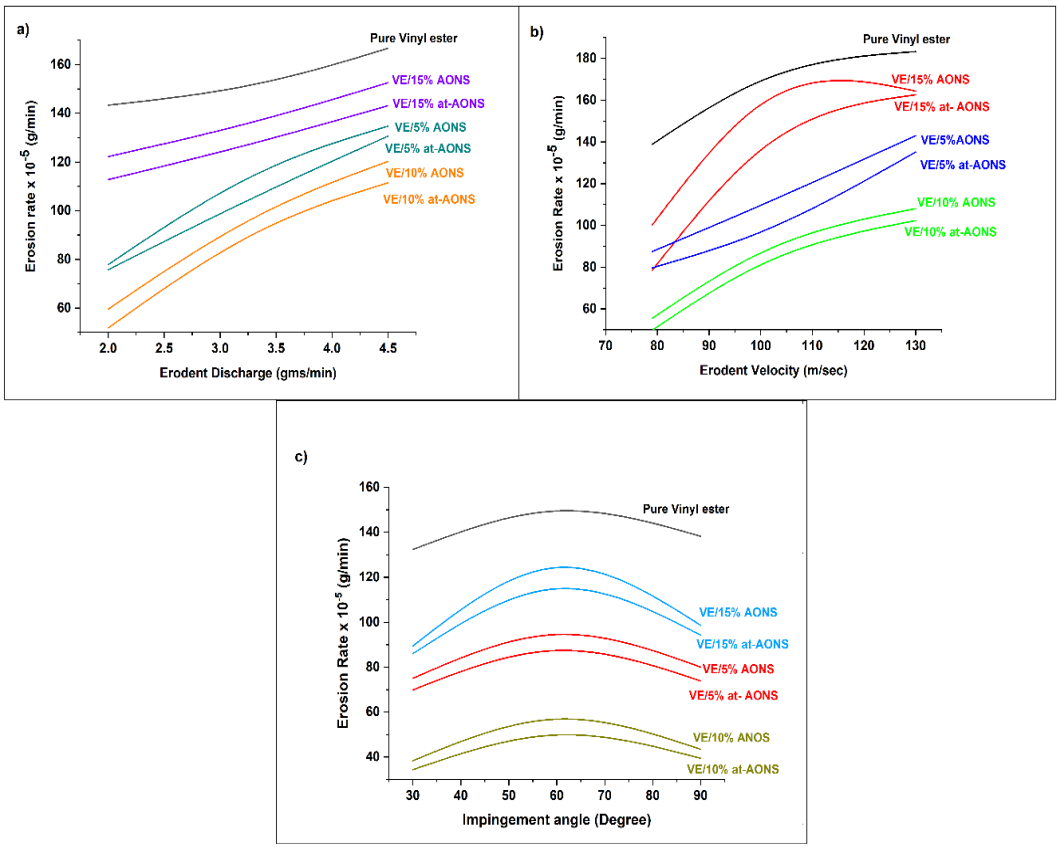

Figure 8 Erosion Rate of composites against (a) Erodent Discharge, (b) Erodent velocity, (c) Impingement angle

According to the graphical representations of this erosion study as shown in Figure 8, the rate of erosion for the untreated AONS Biochar composite was higher than that for the treated AONS biochar composites. It is noteworthy that the treated composites with the lowest extent of erosion were those with a 10% concentration. Strong liaison between the components of the treated composites was shown to be responsible for their better erosion resistance, which also contributed to their greater durability at warmer temperatures [17]. SEM micrographs showed the optimum test conditions for minimal wear and tear include a 30° impingement angle, a 78 m/s erodent velocity, and a 2 g/min discharge rate. These results showed that in both the treated and untreated processes, the 10%Wt concentration of a-t AONS Biochar composites showed the least amount of erosion. It's crucial to remember that the treated AONS biochar loaded composites outperformed the untreated AONS biochar loaded composites in terms of erosive resistance.

8. CONCLUSIONS

This research investigated the effect of untreated and alkali-treated Anacardium occidentale (cashew nut) shell biochar on the physico-chemical, thermal, mechanical, electrical, and surface erosion properties of vinyl ester composites. Based on the findings, the following conclusions are arrived.

- Alkali treatment significantly improved the surface chemistry of the biochar, enhancing its compatibility with the vinyl ester matrix. Treated biochar composites exhibited better dispersion and interaction within the matrix compared to untreated biochar composites, leading to improved overall performance. The O-H, N-H, C-H bonds are increased its wavenumber gradually while adding the 5, 10 and 15wt. % of untreated AONS biochar in vinyl ester matrix. The crystallinity index of 15wt. % AONS biochar loaded composited increased by 31.29% than pure vinyl ester specimen.

- The incorporation of biochar, especially the alkali-treated variant, improved the thermal stability of the vinyl ester composites. Thermogravimetric analysis (TGA) showed that composites with treated biochar had higher decomposition temperatures, indicating enhanced thermal resistance.

- Mechanical testing revealed that composites containing alkali-treated biochar had superior tensile, flexural, and impact strengths compared to those with untreated biochar. The enhanced mechanical properties are attributed to the better interfacial adhesion between the treated biochar and the vinyl ester matrix. The tensile strength of pure vinyl ester plate is 25.9 MPa. VER/10wt. % untreated AONS composites with stand more tensile strength of 43.1 MPa than other combinations.

- Better flexural strength and an increase of 28.87% were demonstrated by 15% of untreated ANOS biochar-incorporated composites compared to pure vinyl ester plates.

- The storage modulus (a measure of the material's stiffness) of vinyl ester composites increased with the addition of biochar, particularly with alkali-treated biochar. This indicates improved stiffness and rigidity due to better filler-matrix interaction. The highest storage modulus for VER/15wt% AONS biochar composites ranges from 2700 MPa to 750 MPa, which is equivalent to a temperature increase of -50 ^{o}C to 175 ^{o}C.

- The incorporation of biochar, especially the alkali-treated variant, resulted in a higher glass transition temperature (Tg) of the composites. This implies improved thermal stability and resistance to softening at elevated temperatures. The increase in Tg is attributed to the restricted polymer chain mobility due to the strong interfacial adhesion between the treated biochar and the vinyl ester matrix. The thermal degradation temperature for pure vinyl ester plate is 310 ^{o}C, VER/15wt. % AONS biochar loaded composites are thermally degraded at 427.5 ^{o}C and VER/15wt. % a-t AONS composites can degrade thermally by 433.4 ^{o}C.

- The electrical conductivity of the vinyl ester composites was influenced by the type of biochar used. Treated biochar composites demonstrated higher electrical conductivity. The 15wt.% of untreated AONS biochar loaded composites only conduct the electricity of 0.025 S/m . The electrical conductivity of 15wt. % of alkali treated AONS biochar loaded vinyl ester composites are 0.041 S/m. This increase in conductivity can be linked to the improved dispersion and connectivity of the biochar particles within the polymer matrix.

- Surface erosion tests indicated that alkali-treated biochar composites had better erosion resistance compared to untreated biochar composites. The enhanced erosion resistance is due to the stronger matrix-filler interaction in treated biochar composites, which helps in maintaining the integrity of the composite surface under erosive conditions.

From the results it was absorbed that the alkali-treated Anacardium occidentale nut shell biochar is a promising filler material for enhancing the performance of vinyl ester composites. The treatment process significantly improves the interfacial properties between the biochar and the polymer matrix, leading to composites with superior thermal, mechanical, electrical, and surface erosion properties compared to those incorporating untreated biochar. These findings suggest potential applications in fields requiring materials with high strength, thermal stability, electrical conductivity, and erosion resistance.

9. ORGANIZATION OF THESIS

Chapter 1: This chapter discuss about the classifications of composite materials and different types of polymer resins. The various types of reinforcements such as natural fibers and biochar also discussed. This chapter also elaborated the functional applications of biochar and also importance of surface modification on biochar to fabricate the high compatible polymer composites.

Chapter 2: This chapter contains comprehensive literature review on effect of reinforcement fillers in polymeric composites. The functional properties of composites plates such as mechanical behaviour, thermal behaviour, electrical conductivity, viscoelastic properties and erosion properties of natural fibers / particulate fillers reinforced polymeric composites has been described. This chapter also labelled the various types biochars and its characterization properties with polymeric resins. The research gap was found and the intention of their research study was given in accordance with the literature review.

Chapter 3: This chapter describe the materials, fabrication methodology and experimental methods for the present work. The accessories needed and resizing of materials for various characterization techniques as per ASTM standards also discussed. The main objective of this thesis is comparison of effect of various

concentrations (%5, 10% &15wt. %) of raw and alkali treated cashew (Anacardium occidentale) nut shell biochar in characteristic properties of vinyl ester based composites.

Chapter 4: This chapter briefly elucidates the physiochemical properties of vinyl ester resin and raw and alkali treated cashew (Anacardium occidentale) nut shell biochar. The FTIR, XRD, TGA, DTG, Tensile, Flexural, Impact testing for pure vinyl ester specimens were discussed. The particle size analysis, elemental composition. X- Ray diffraction, Fourier Transform Infra-Red (FT-IR) Spectroscopy analysis, surface morphology, thermal decomposition and electrical conductivity of raw and alkali treated AONS biochar also illustrated.

Chapter 5: This chapter discuss about the effect of different proportions (5%, 10% and 15wt. %) of untreated and alkali treated anacardium occidentale nut shell (AONS) biochar on Physio-chemical, of vinyl ester based composites was reported.

Chapter 6: This chapter discuss about effect of different proportions (5, 10 and 15wt. %) of untreated and alkali treated anacardium occidentale nut shell (AONS) biochar on thermal and viscoelastic properties of vinyl ester based composites was reported.

Chapter 7: This chapter discuss about effect of different proportions (5, 10 and 15wt. %) of untreated and alkali treated anacardium occidentale nut shell (AONS) biochar on Tensile, flexural and impact properties of vinyl ester based composites was described. The effect of AONS biochar on the electrical conductivity of all the fabricated composites was discussed. Further, failure analysis of composites against tensile strength, flexural strength and impact strength also analysed by using SEM images of fractured surface.

Chapter 8: This chapter presents about the solid particle erosion properties of VER/AONS and VER/a-t AONS composites. The erosion rate of all the samples were analysed based on erodent discharge, erodent velocity and impingement angle.

Chapter 9: The conclusions of the present research work and the scope of future investigation for this study is described in this chapter.

REFERENCES

1. R. Sundarakannan, V. Arumugaprabu, V. Manikandan, and R.D.J. Johnson, (2020). Tribo performance studies on redmud filled pineapple fiber composite, *Materials Today Proceedings,* **24** (2020), 1225-1234.
2. P.U. Rani, B.M. Rajaprakash, N. Mohan and M.A. Prasad, Study on thermal and erosive wear behaviour of hard powders filled glass-epoxy composite, *Materials Today Proceedings*, **27** (2020), 2011-2016.
3. A.Y. Elnour, A.A. Alghyamah, H.M. Shaikh, A.M. Poulose, S.M. Al-Zahrani, A. Anis, A and M.I. Al-Wabel, Effect of pyrolysis temperature on biochar microstructural evolution, physicochemical characteristics, and its influence on biochar/polypropylene composites, *Applied sciences*, **9** (6) (2019), 1149.
4. O. Das, N. K. Kim, M. S. Hedenqvist, R. J. Lin, A. K. Sarmah and D. Bhattacharyya, an attempt to find a suitable biomass for biochar-based polypropylene bio composites, *Environmental management*, **62** (2018), 403-413.
5. R.Sundarakannan, V. Arumugaprabu, V. Manikandan and S. Vigneshwaran, Mechanical property analysis of biochar derived from cashew nut shell waste reinforced polymer matrix, *Materials Research Express*, **6** (12) (2020), 125349.
6. S. Richard, J. Rajadurai and V. Manikandan, Effects of particle loading and particle size on tribological properties of biochar particulate reinforced polymer composites, *Journal of Tribology*, **139** (1) (2017), 012202.
7. M. Giorcelli, P.Savi, A. Khan and A. Tagliaferro, Analysis of biochar with different pyrolysis temperatures used as filler in epoxy resin composites, *Biomass and bioenergy*, 122 **(**2019), 466-471.
8. D. Ray, B.K. Sarkar, R. K. Basak and A.K. Rana, Thermal behavior of vinyl ester resin matrix composites reinforced with alkali-treated jute fibers, *Journal of applied polymer science*, **94** (1) (2004), 123-129.
9. M. Thiruchitrambalam, A. Athijayamani, S. Sathiyamurthy and A.S.A. Thaheer, A review on the natural fiber-reinforced polymer composites for the development of roselle fiber-reinforced polyester composite, *Journal of Natural Fibers*, **7** (4) (2010), 307-323.

10. P. Sivamurugan, M. Mareeswaran, S.M. Abraar, S. Verma, N. Verma, B.K Srivastava and B. Ramesh, Development of thermally reduced corn stover biochar and its satin weaved sisal-reinforced vinyl ester composites, *Biomass Conversion and Biorefinery*, (2023) 1-9.

11. K. Ramraji, K. Arunkumar, R. Senthilkumar, M. Rajesh, and A.J.P. Kumar, Mechanical and thermomechanical behaviour of agro-waste almond shell biochar filler interlaced chemically treated flax fibre vinyl ester composites, *Biomass Conversion and Biorefinery*, (2023) 1-14.

12. O. Das, N. K. Kim, M. S. Hedenqvist, R. J. Lin, A. K. Sarmah and D. Bhattacharyya, an attempt to find a suitable biomass for biochar-based polypropylene biocomposites, *Environmental management*, **62** (2) (2018), 403-413.

13. P. S. Latha and M. V. Rao, Investigation into effect of ceramic fillers on mechanical and tribological properties of bamboo-glass hybrid fiber reinforced polymer composites, *Silicon*, **10** (4) (2018), 1543-1550.

14. R. O. Akaluzia, F. O. Edoziuno, A. A. Adediran, B. U. Odoni, S. Edibo and T. M. A. Olayanju, Evaluation of the effect of reinforcement particle sizes on the impact and hardness properties of hardwood charcoal particulate-polyester resin composites, *Materials Today Proceedings*, **38** (2021), 570-577.

15. A. Pudełko, P. Postawa, T. Stachowiak, K. Malinska and D. Drozdz, Waste derived biochar as an alternative filler in bio composites-Mechanical, thermal and morphological properties of biochar added bio composites, *Journal of cleaner production*, **278** (2021), 123850.

16. O.P. Minugu, R. Gujjala, O. Shakuntala, P. Manoj and M. S. Chowdary, Effect of biomass derived biochar materials on mechanical properties of biochar epoxy composites, *Proceedings of the Institution of Mechanical Engineers, Part C Journal of Mechanical Engineering Science*, **235** (21) (2021), 5626-5638.

17. A. Akhtar and A. K. Sarmah, Novel biochar-concrete composites, Manufacturing, characterization and evaluation of the mechanical properties, *Science of the total environment*, **616** (2018), 408-416.

PUBLICATIONS

1. **R. Pradeep**, V. Arumugaprabu, P. Geetha, R. Sundarakannan, S. Vigneshwaran, K. Naresh and M. Uthayakumar, The development and multifunctional characterization of cashew (Anacardium occidentale) nut shell biochar reinforced vinyl ester composites for sustainable management, *Biomass Conversion and Bio refinery*, (2023), 1-14.

2. **P. Rajamani**, A. Veerasimman, G. Palani, V. Shanmugam, S. Rajendran, A. Subbaiah and R. GB, Enhancing vinyl ester properties with eco-friendly sustainable biochar filler, *Polymer Composites*, **44** (12) (2023), 8344-8352.

3. **R. Pradeep**, A. Veerasimman and S. Rajendran, A comparative study on erosion behaviour of untreated and alkaline-treated biochar reinforced vinyl ester composites, *Interactions*, **245** (1) (2024), 92.